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APPLICANT : NKK CORP;

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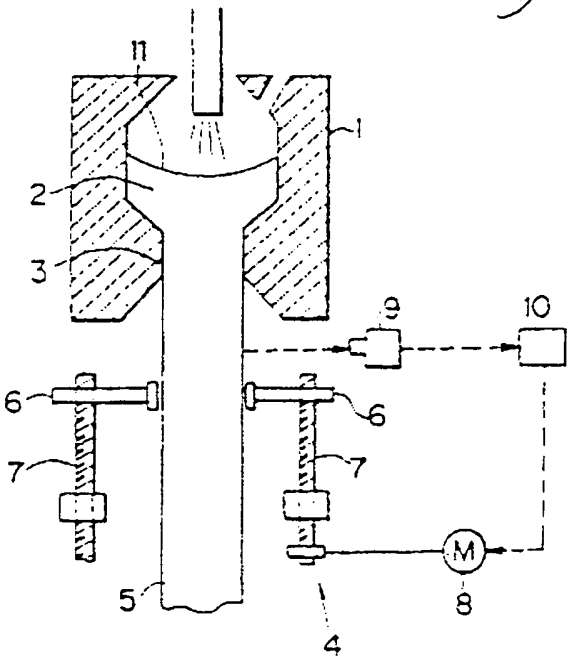
TITLE : PRODUCTION OF SILICA



ABSTRACT : PURPOSE: To stably produce a superior-quality silica rod by controlling the drawing velocity of the silica rod which is drawn out through a squeeze part of the lower part of a furnace and holding the temp. of the surface of the silica rod in the lower part of the squeeze part constant.

CONSTITUTION: A silica rod 5 is continuously obtained by drawing silica 2 melted in a furnace 1 with a drawer 4 through a squeeze part 3 formed to the lower part of the furnace 1 and solidifying it by cooling in air. The drawer 4 is constituted of bands 6 for inserting the rod 5 between and screw rods 7 which are rotated with a motor 8 and vertically move the bands 6. In the above-mentioned method, the temp. of the surface of the rod 5 in the lower part of the squeeze part 3 is measured with a two-color thermometer and the measured value is sent to a controlling device 10. Therein the drawing velocity of the rod 5 is controlled via the motor 8 so that the temp. of the surface is regulated to the previously preset target temp.

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Specification

1. Title of Invention

Method of manufacturing silicic acid

2. Claim

A method of preparing silicic acid characterised in that silicic acid is manufactured continuously by drawing a silicic acid rod from a die part formed at the bottom of the furnace wherein the silicic acid is melted, and in that the surface temperature of the said silicic acid rod is measured at the bottom of the die part and the rate at which the silicic acid rod is drawn is adjusted so that this surface temperature remains constant.

3. Detailed Description of the Invention

[Area of industrial utility]

The present invention relates to a method of manufacturing silicic acid rod continuously.

(Prior art)

Silicic acid rod prepared by melting raw materials such as silica and quartz in a furnace are used as IC sealants and also as fire-resistant materials, high-strength glass, lost wax encasing, catalysts and cosmetics. There is a particular requirement for very high quality products for IC sealants. Because of this, in methods of silicic acid rod production of the prior art, an important factor for the improvement of quality was the maintenance of an appropriate retention time of the silicic acid in the furnace.

In order to maintain the retention time in the furnace constant, in the prior art, as shown in Figure 4, an operative measures by eye, through measurement aperture 20, the height and vertical variations of the melt surface 11 of the molten silicic acid 2 in the furnace 1 and, using the drawing device 4, changes the rate at which the silicic acid rod 5 is drawn from the die part 3 at the bottom of the furnace on the basis of these observations to maintain the melt surface level 11 and keep the retention time constant. However, it is difficult to keep the height of the melt surface11 steady by such visual measurement and the retention time varies through a range.

In another method, specified in JP 54-21412 (A), as shown in Figure 5, the surface of silicic acid rod 5 descending from the die part 3 at the bottom of the furnace 1 is either heated by a burner or cooled by water from a cold water pipe 21 to keep the temperature at 1200°C~1600°C and maintain the rate of vertical movement of the rod constant and thus maintain the retention time of the silicic acid in the furnace constant.

(Problems the invention aims to solve)

However, the above methods have the following problems.

(1) In the method in which the height of the melt surface is measured by eye and the rate at which the silicic acid rod is drawn is adjusted accordingly:

When the melt surface falls below the mark, and the rate at which the silicic acid rod is drawn is too fast, the retention time of the silica acid is too short and the rod produced has an admixture of unmelted material, resulting in quality deterioration. Also, if the rate of drawing is too fast, the cooling is inadequate so that the silicic acid rod remains soft and may bend or thicken, abrading and damaging the bricks of the die part. Fragments of these bricks may then melt on to the surface of the silicic acid rod, lowering its quality.

When the level of the melt surface rises, and the rate of drawing is too slow, the rod being drawn is cooled excessively so that its temperature is too low and it loses viscosity. In this state it adheres to the bricks at the die part at the bottom of the furnace and damages them so that fragments of these bricks may then melt into the silicic acid rod, lowering its quality. Also if the temperature of the drawn silicic acid rod falls below a certain point, it becomes impossible to draw and fractures, necessitating an interruption of the production process.

Thus the problem involved in this method is deterioration in quality when the rate at which the silicic acid rod is drawn is either too fast or too slow.

(2) When a method in which the silicic acid rod is heated or cooled at the bottom part of the furnace is used:

In this method the temperature of the silicic acid rod at the die part outlet of the bottom of the furnace is controlled to maintain the descent of the rod at a fixed rate, thus apparently solving the problems involved in the above method. However, the requirement for heating and cooling equipment imposes an energy consumption penalty.

The present invention has the object of proposing a method of producing silicic acid rod which solves the above problems and allows stable production of silicic acid rod of good quality.

[Means by which the problems are solved]

Thus the present invention is a method of preparing silicic acid characterised in that silicic acid is manufactured continuously by drawing a silicic acid rod from a die part formed at the bottom of a furnace and in that the surface temperature of the said silicic acid rod is measured at the bottom of the said die part and the rate at which the silicic acid rod is drawn is adjusted so that this surface temperature remains constant.

[Actions]

In standard operation, when silicic acid rod is produced continuously, a constant quantity of raw material is fed continuously and a constant quantity of fuel, used to melt the raw materials, is also fed continuously so that the temperature of the molten silicic acid in the furnace is kept constant.

Accordingly, the temperature of the silicic acid rod drawn from the bottom of the furnace is determined by the rate at which the rod is drawn. Therefore, if the drawing speed is too fast, the temperature of the silicic acid rod drawn from the die part at the bottom of the furnace is high, since the cooling during drawing is inadequate while, conversely, if the drawing speed is too slow, the cooling is more effective and the temperature of the silicic acid rod is lower. In the method according to the invention, the surface temperature of the silicic acid rod is measured at the bottom of the said die part and the drawing rate of the silicic acid rod

controlled so that this temperature is kept constantly at an appropriate level. This keeps both the drawing rate and melt surface height constant. Because of this the molten silicic acid in the furnace is kept in an optimal condition.

[Examples]

Below exemplary embodiments of the invention are described with reference to figures. Figure 1 is a sectional figure showing one embodiment of equipment to carry out the method according to the invention. In this, molten silicic acid 2 in furnace 1 is drawn by drawing device 4 from die part 3 formed at the bottom of furnace 1 and this cools and hardens to form the silicic acid rod 5.

The said drawing device 4 consists essentially of a screw-threaded shaft which, by rotating, raises and lowers the supports 6 which support the silicic acid rod 5 and a motor 8 which rotates screw-threaded shaft 7. This drawing device is located in two stages, upper and lower, (the lower drawing device is not shown) and the upper and lower drawing devices operate reciprocally to draw the silicic acid rod continuously. The surface temperature of the silicic acid rod 5 drawn from the die part 3 at the bottom of the furnace 1 is constantly monitored by a dichromatic thermometer 9. The temperatures thus measured are converted into electronic signals and sent to control device 10. This control device 10 increases or reduces the rpm of motor 8 of drawing device 4 according to the difference between these signals and a pre-set temperature and thus controls the speed at which the silicic acid rod 5 is drawn. Accordingly, the ~~metal~~ melt surface height 11 of the silicic acid 2 in the furnace 1 is kept steady. Since the silicic acid rod 5 is drawn at a steady temperature and the height of the melt surface 11 of the molten silicic acid 2 in the furnace 1 is kept steady, so the retention time of the silicic acid in the furnace 1 is kept constant making it possible to produce, stably and continuously, silicic acid rod 5 of high quality containing no admixture of unmelted silicic acid, and with no shards of the bricks of the die part 3 at the bottom of furnace 1 melted on to its surface.

In order to make a comparison between the preparation method according to the invention and the prior art, silicic acid rod was prepared using the equipment shown in Figure 1 and that shown in Figure 4. Figures 2 (a)~(c) are explanatory figures showing changes in the surface temperature, drawing rate and melt surface height in the method of manufacture according to the invention. Figures 3 (a)~(c) are explanatory figures showing the said values in the method of the prior art. As is clear from Figures 2 and 3, whereas these values showed a wide range of variation in the method of the prior art, they were remarkably stable in the method according to the invention, with the surface temperature of the silicic acid rod remaining approximately stable near 1450°C. Because of this, the melt surface level remained stable and with a range of variation about one-fifth of that in the method of the prior

art.

Table 1 shows the incidence of unmelted silicic acid raw material, found from the surface temperature of the silicic acid rod and the incidence of stoppages of furnace operation due to fracture of the silicic acid rod during drawing.

As is clear from this table, the method according to the invention shows markedly lower incidences of unmelted raw material and stoppages than the method of the prior art.

Table 1

	Unmelted material incidence	Stoppage incidence
Prior art	30 times/year	12 times/year
Invention Temperature 1500~1550°C	10	1
1400~1500°C	2	2
1300~1400°C	5	5

(Effects of the invention)

When the method according to the invention is used, the drawing rate is controlled so that the surface temperature of the drawn silicic acid rod remains steady and this means that not only is it possible to keep the surface temperature at an optimal level but also maintain the melt surface height and thus produce high-quality silicic acid rod stably and continuously.

4. **Simple Description of the Figures**

Figure 1 is a sectional figure showing one embodiment of the equipment used for the method according to the invention. Figures 2 (a)~(c) show the operational results of the method according to the invention. . Figures 3 (a)~(c) show the operational results of methods of the prior art. Figures 4 and 5 are sectional figures showing embodiments of equipment used for the method of the prior art.

1 ... furnace; 2 ... molten silicic acid; 3 ... die part; 4 ... drawing device;

5 ... silicic acid rod; 8 ... motor; 9... dichromatic thermometer; 10 ... control equipment.

Patent applicant: Japan Copper Tubes Company Limited

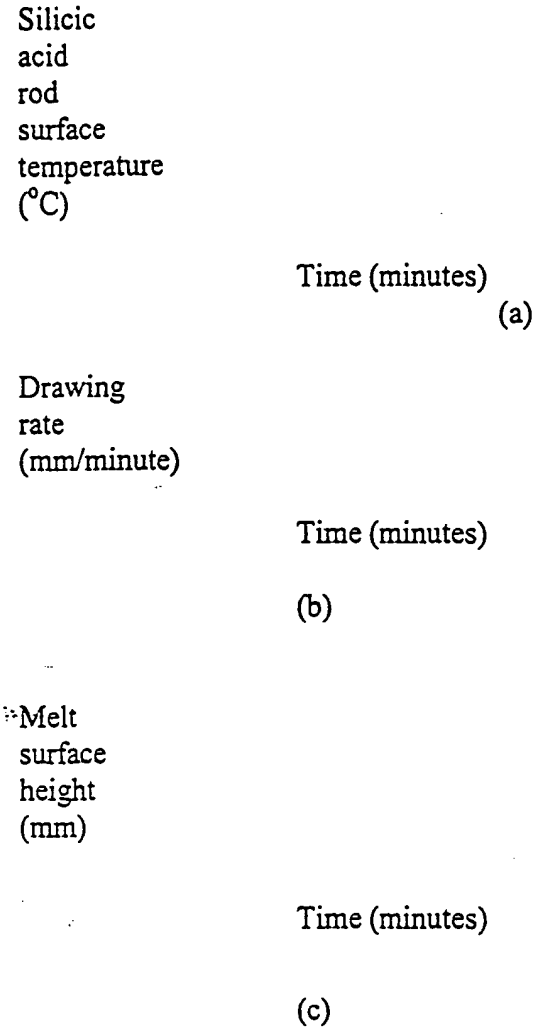

Figure 3

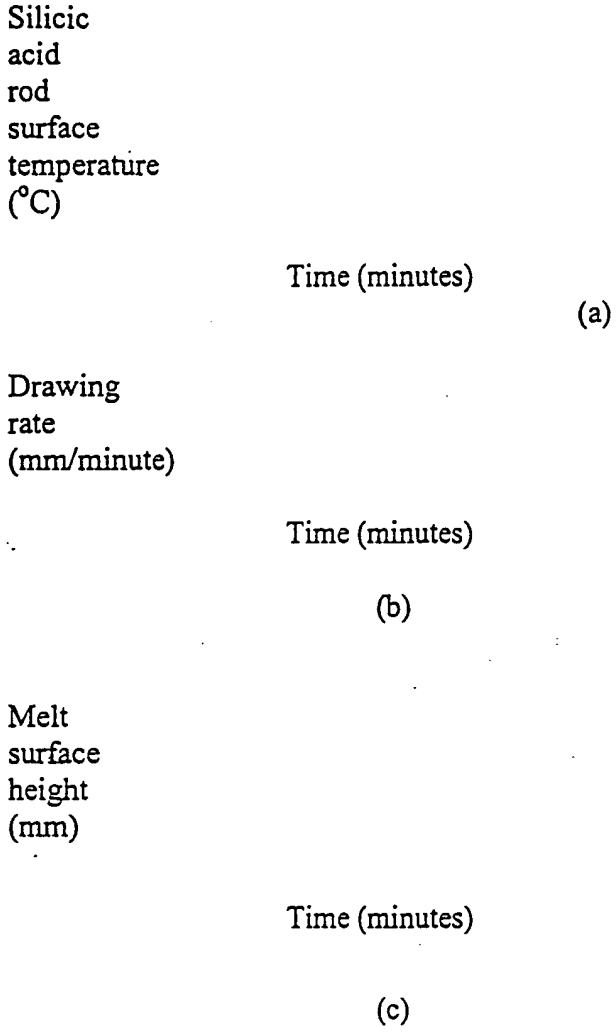

Figure 2

Visual measurement

Figure 4

PRODUCTION OF SILICA

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Abstract

PURPOSE:To stably produce a superior-quality silica rod by controlling the drawing velocity of the silica rod which is drawn out through a squeeze part of the lower part of a furnace and holding the temp. of the surface of the silica rod in the lower part of the squeeze part constant.
CONSTITUTION:A silica rod 5 is continuously obtained by drawing silica 2 melted in a furnace 1 with a drawer 4 through a squeeze part 3 formed to the lower part of the furnace 1 and solidifying it by cooling in air. The drawer 4 is constituted of bands 6 for inserting the rod 5 between and screw rods 7 which are rotated with a motor 8 and vertically move the bands 6. In the above-mentioned method, the temp. of the surface of the rod 5 in the lower part of the squeeze part 3 is measured with a two-color thermometer and the measured value is sent to a controlling device 10. Therein the drawing velocity of the rod 5 is controlled via the motor 8 so that the temp. of the surface is regulated to the previously preset target temp.

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C 03 B 17/04		7344-4G	審査請求 未請求 発明の数 1 (全4頁)

㊹発明の名称　ケイ酸の製造方法

㉑特　願　昭62-154430

㉒出　願　昭62(1987)6月23日

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明　細　書

1．発明の名称

ケイ酸の製造方法

2．特許請求の範囲

炉内で溶融されたケイ酸を炉下部に形成された絞り部から引抜きケイ酸ロッドを連続的に製造する方法において，前記絞り部の下部における前記ケイ酸ロッドの表面温度を測定し，この表面温度が一定になるように前記ケイ酸ロッドの引抜き速度を制御することを特徴とするケイ酸の製造方法。

3．発明の詳細な説明

〔産業上の利用分野〕

本発明はケイ酸ロッドの連続的製造方法に関する。

〔従来の技術〕

珪砂や珪石の原料を炉内で溶融して製造したケイ酸ロッドはICの封止材をはじめとして，耐火物，高級ガラス，ロストワックスの衣，触媒，化粧品等に使用される。そして，特にICの封止材として使用される場合には非常に高品質のものが要求される。このため，ケイ酸ロッドの製造においては，その品質を高める要件の一つとして，炉内におけるケイ酸の滞留時間を適正に保つことが重要な管理項目として挙げられている。

炉内におけるケイ酸の滞留時間を一定にするために，従来の技術では，第4図に示すように作業者が炉1内の溶融ケイ酸2の湯面11の高さや湯面が上下する変動を測定孔20から目視測定し，この結果に基づいて，炉1下部の絞り部3からケイ酸ロッド5を引抜く引抜き装置4の引抜き速度を変えて，湯面11を一定の高さに保持し，滞留時間が一定になるようにしていた。しかしながら，このような目視による測定では湯面11を一定の高さに保つことは困難であり，従って，滞留時間もある幅をもって変動していた。

また同様の目的のために特開昭54-21412号公報においては，第5図に示すように，炉1下部の絞

り部3から降下するケイ酸ロッド5の表面をバーナーあるいは冷却用水[redacted]により加熱，冷却して1200℃～1600℃に保持してケイ酸ロッドの降下速度を一定にし，炉1内のケイ酸の滞留時間を一定に保持している。

〔発明が解決しようとする問題点〕

しかしながら，上述した従来の技術には次のような問題点があった。

(1) 湯面の高さを目視測定してケイ酸ロッドの引抜き速度を変える方法の場合

湯面の高さが目視より低下した時は，ケイ酸ロッドの引抜き速度が速すぎる場合であり，このような際にケイ酸の炉内での滞留時間が短いので未溶融の原料が混入した状態で引き抜かれてケイ酸ロッドが形成され，品質の低下を招く。また引抜き速度が速すぎると冷却が不十分の状態であるので，ケイ酸ロッドはまだ軟らかく，曲がったり，肥大化して炉下部の絞り部の煉瓦をこすって損傷させ，この煉瓦片がケイ酸ロッドの表面に溶着して品質の低下となる。

湯面の高さが上昇した時に，ケイ酸ロッドの引抜き速度が遅すぎ[redacted]合であり，このような際は引抜かれるケイ酸が冷却されすぎて温度が低下し，粘性がない状態になってしまうので，炉下部の絞り部の煉瓦と固着して煉瓦を損傷するとともにこの損傷した煉瓦がケイ酸ロッドに溶着して品質を低下させる。また引抜くケイ酸ロッドの温度がある値以下に低下すると引抜き不能あるいは破断が発生し，操業を停止しなければならなくなる。

このようにケイ酸ロッドの引抜き速度が速すぎても，遅すぎても品質の低下をはじめ種々の問題が発生する。

(2) 炉下部においてケイ酸ロッドを加熱，冷却する方法の場合

この方法は炉下部の絞り部出口におけるケイ酸ロッドの温度を制御してケイ酸ロッドの降下速度を一定にするものであり，上記の問題点を一応解決したものであるが，加熱，冷却装置を必要としてエネルギーの無駄な消費がある。

本発明は以上のような従来技術の問題点を解消

し，品質のよいケイ酸ロッドを安定して生産できる方法を提供することを目的とする。

〔問題点を解決するための手段〕

本発明は炉内で溶融されたケイ酸を炉下部に形成された絞り部から引抜きケイ酸ロッドを連続的に製造する方法において，前記絞り部の下部における前記ケイ酸ロッドの表面温度を測定し，この表面温度が一定になるように前記ケイ酸ロッドの引抜き速度を制御することを特徴とする。

〔作　用〕

ケイ酸ロッドを連続的に製造する場合の定常操業においては，定量の原料を連続的に供給し，また原料を溶融する燃料も一定量を供給して炉内の溶融ケイ酸の温度を一定に保持している。

従って，炉下部から引抜かれたケイ酸ロッドの温度はその引抜き速度によって決まる。すなわち引抜き速度が速ければ，引抜きの間における冷却が不十分のため炉下部の絞り部から引抜かれたケイ酸ロッドの温度は高くなり，逆に引抜き速度が遅ければ冷却がよく行われるのでケイ酸ロッドの

温度は低くなる。本発明では前記絞り部の下部におけるケイ酸ロッドの表面温度を測定し，この表面温度が適温であり且つ一定になるようにケイ酸ロッドの引抜き速度を制御するので，引抜き速度は一定になり，従って湯面の高さも一定に維持される。このため炉内のケイ酸の溶融状態は常に最適状態に保たれる。

〔実施例〕

以下，本発明の実施例について説明する。第1図は本発明を実施する装置の一実施例を示す断面図である。炉1内で溶融された溶融ケイ酸2は炉1下部に形成された絞り部3から引抜装置4によって引抜かれ，放冷によって固形ケイ酸ロッド5となる。

前記引抜装置4の要部はケイ酸ロッド5を挟持するバンド6と回転することによりバンド6を上下させる螺杆7及び螺杆7を回転させるモーター8とで構成されている。この引抜き装置4は上下に2段配置され（下段の引抜き装置は図示せず），上下の引抜き装置が交互に作動して，ケイ酸ロッ

て，炉1下部の絞り部3から引抜かれたケイ酸ロッド5の表面温度を2色温度計9によって常時測定するようにしてある。測定された温度は電気信号に変換されて制御装置10に送られる。この制御装置10にあらかじめ設定されている目標温度との差に応じて引抜き装置4のモーター8の回転数を増減させてケイ酸ロッド5の引抜き速度を制御する。このため，ケイ酸ロッド5の引抜き速度は一定になり，従って炉1内の溶融ケイ酸2の湯面11も一定に保たれる。このようにして，ケイ酸ロッド5が一定の温度で抜き出され，且つ溶融ケイ酸2の湯面11が一定に保たれることにより炉1内でのケイ酸の滞留時間も一定となるので，ケイ酸ロッド5に未溶融のケイ酸が混入することもなく，炉1下部の絞り部3の煉瓦がケイ酸ロッドに溶着することもなくなり，高品質のケイ酸ロッド5を安定して連続的に製造することができる。

次に本発明の製造法と従来技術とを比較するために第1図の装置と第4図の装置を使用してケイ

第1表

	未溶融頻度	炉停止頻度
従来の技術	30回／年	12回／年
本発明　温度1500～1550℃	10　〃	1　〃
1400～1500℃	2　〃	2　〃
1300～1400℃	5　〃	5　〃

〔発明の効果〕

本発明によれば，引き抜いたケイ酸ロッドの表面温度が一定になるように引抜き速度を制御しているので，その表面温度を適温に保持できるとともに湯面も一定に維持することができ，高品質のケイ酸ロッドを安定して連続的に製造することができる。

4．図面の簡単な説明

第1図は本発明を実施する装置の一実施例を示す断面図，第2図(a)～(c)は本発明の方法による操業実績の説明図，第3図(a)～(c)は従来技術による操業実績の説明図，第4図及び第5図は従来技術

ケイ酸ロッドを製造した操業実績について説明する。第2図(a)～(c)は本発明の製造法におけるケイ酸ロッドの表面温度，引抜き速度，湯面の高さについての変動を図示した説明図であり，第3図(a)～(c)は従来技術の製造法における前記の測定値を図示した説明図である。第2図及び第3図で明らかなように，従来技術においては大幅に変動している各測定値が本発明では非常に安定し，ケイ酸ロッドの表面温度は1450℃付近で略一定になっている。このため湯面の高さも安定し，その変動幅は従来技術の場合の1/5 程度になっている。

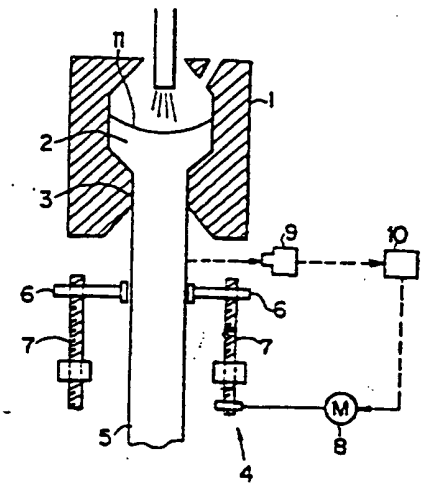
また第1表はケイ酸ロッドの表面温度によるケイ酸原料の未溶融の発生頻度及び引抜き時にケイ酸ロッドが破断してしまったために炉の操業を停止した頻度の実績を示したものである。

この表で明らかなように，本発明は従来技術に比較し，未溶融および炉停止の頻度が著しく減少している。

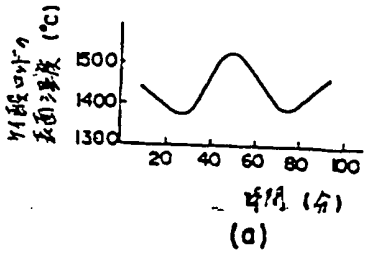
において使用する装置の断面図である。

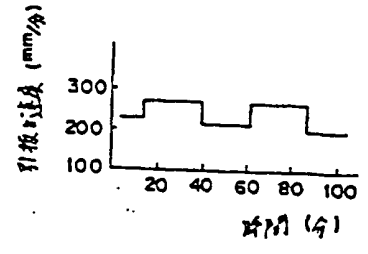
1…炉，　2…溶融ケイ酸，　3…絞り部，
4…引抜き装置，　5…ケイ酸ロッド，
8…モーター，　9…2色温度計，
10…制御装置

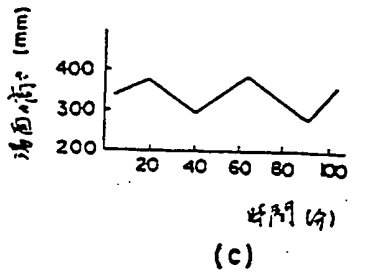
特許出願人　　日本鋼管株式会社



第1図







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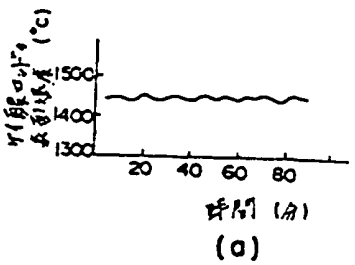

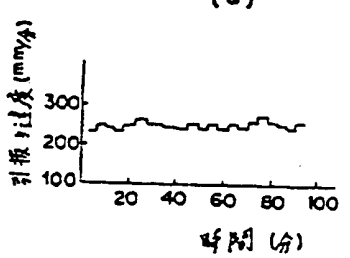

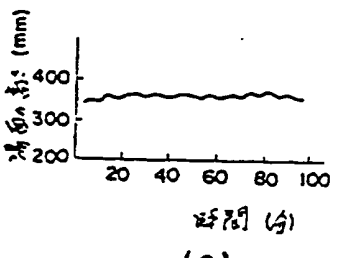

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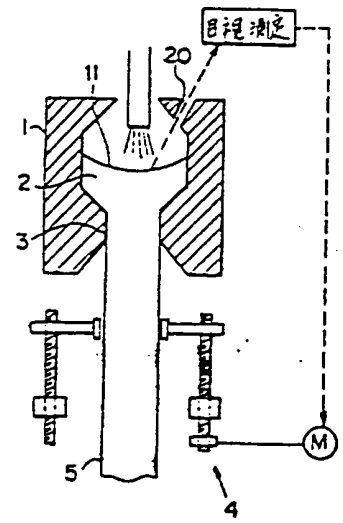
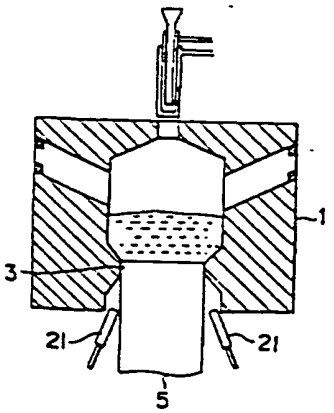


第4図



第5図